

Ultrathin materials formed from fiber and superabsorbent

Description

5 The present invention relates to materials formed from superabsorbent polymer (SAP) and fibers that are obtainable by pressing at not less than 60°C and not less than 3 bar. More particularly, the present invention relates to materials which are obtained by in situ polymerization of SAP precursor mixtures on the fiber. The present invention also relates to processes for producing such materials and to their use.

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In situ materials have been known since the early 1980s. They are notable for a sheetlike fibrous structure, especially a nonwoven, being treated with liquid medium which, after polymerization on the fibrous structure (in situ), forms an absorbent polymer. Polymerization may be initiated in any known way, as by radiation (UV, electron beam, heat), additives (eg redox initiators). The liquid medium contains monomers and possibly comonomers for forming the absorbent polymer.

15 Crosslinkers, further optional additives eg odor inhibitors, thickeners, SAP fine powder, etc may already be present in the liquid medium or only added to it after the treatment of the sheetlike structure. An aftertreatment after the polymerization, for example surface postcrosslinking, can follow. The treatment of the sheetlike structure with the liquid medium can be accomplished by

20 spraying, drenching or other customary treatment methods.

In situ materials are known in the prior art for example in EP 40 087, EP 54 841, EP 123 500, EP 108 637, EP 223 908, EP 315 185, WO 95/33878, WO 01/56625.

25 Materials formed from SAP and fibers by admixing the SAP to the fibers are known. This can be accomplished in various ways, for example by addition of SAP to the process for producing a sheetlike fibrous material (air laid or wet laid) or by addition of the SAP after the fibrous material has already been shaped into a sheetlike structure. The SAP can then be fixed to the fibers by various methods, for example adhesion agents. The SAP can also be embedded as a layer

30 between two fibrous layers (see for example: WO 95/30396).

There are many applications in the hygiene sector and non-hygiene applications where aqueous fluids are to be absorbed where it would be desirable to have materials which have one or preferably more than one of the following properties: essentially on contact with liquid to expand in

35 one direction only, to be present in compressed form to minimize storage and transportation costs and to retain shape during storage, which have a high absorbency for aqueous solutions, for example measured in the teabag test, which provide fast liquid acquisition without pressure and under pressure, which are suitable for use as a component of laminates.

40 It has been found that, surprisingly, material formed from SAP and fibers that is obtainable by

pressing at not less than 60°C and not less than 3 bar has the desired properties.

Compression by the action of pressure to produce "ultrathin" hygiene articles is described in WO 01 / 56625. However, the material is subjected to a pressure of about 5.5 bar (fabric area: 0.056 m²; 7.000 pounds load) and a temperature of 50°C for a period of 48 seconds. This achieves a compression from originally 4.5 mm to 0.67 mm. These experimental conditions were reproduced and two differences and disadvantages compared with the present invention were ascertained:

- a) the material is not dimensionally stable, ie it expands to as much as 1.5 mm over 2 weeks and to as much as 2.4 mm over 8 weeks.
- b) The method described in the present invention makes it possible to produce significantly thinner, yet very flexible materials than the material described in WO 01 / 56625.

By application of heat and pressure it is possible to compress materials formed from SAP and fibers, for example SAP-nonwoven composites which can be produced according to WO 01 / 56625.

The compression takes place in that dimension in which pressure is applied. The other two dimensions are left substantially unchanged by the process of compression.

Compression can be effected by initially heating the material to the requisite temperature and then applying pressure; similarly, the material can first be subjected to an applied pressure before it is heated to the requisite temperature; preferably, however, the material is compressed by the simultaneous application of pressure and heat.

The material may be compressed not only batchwise – using presses for example – but also continuously – using calenders for example.

It can be demonstrated in the context of the present invention that an application of pressure and heat is one way to obtain materials having the desired properties. The material properties are significantly more affected by varying the temperature than by varying the pressure. It has also been determined that even relatively low pressures from 3 bar, eg 3 bar, 3.5 bar, 4 bar or 4.5 bar are sufficient to produce the novel materials. Preference is given to pressures of 5 or more bar, eg 5, 5.5, 6, 6.5, 7, 7.5, 8, 8.5, 9, 9.5 bar and particular preference is given to pressures above 10 bar, eg 10, 11, 12, 13, 14, 15, 20, 25, 30, 35, 40, 45, 50 or more bar. Pressures of more than 100 bar, in contrast, generally do not lead to a further improvement in material properties. Temperature for the purposes of this invention is the temperature in the

material pressed or to be pressed. When comparatively long residence times are employed for the pressing operation (1 minute for example), the temperature in the material will be substantially equal to the temperature on the surface of the press. Temperatures below 60°C are generally not sufficient. In general, temperatures of 60°C or more are employed, for example 5 60°C or 65°C. Preference is given to temperatures of 70°C or more, for example 70°C, 75°C, especially of 80°C or more, eg 80°C, 85°C, 90°C, 95°C, 100°C or more. The maximum temperature is dependent on the residence time of the material at the temperature, since thermal degradation of the material should be avoided. The optimum temperature range is between 80°C and 180°C, for example 110°C, 120°C, 130°C, 140°C, 150°C, 160°C or 170°C as 10 well as the temperatures recited above. At temperatures above 200°C, the properties may deteriorate.

The desired material properties are achievable after very short times of applying pressure and heat. Residence times of 1 minute are generally sufficient. Longer residence times are generally 15 not harmful, but not desirable for economic reasons. Typical residence times are 10 seconds, 20 seconds, 30 seconds, 40 seconds, 50 seconds or 60 seconds. But is it also possible to make the pressing time shorter for industrial fabrication, ie to use pressing times of 1, 2, 3, 4, 5, 6, 7, 8, 9 or 10 seconds for example. Short pressing times are used with the comparatively higher temperatures. The short residence times are responsible for the fact that the compressed 20 material can be produced continuously, using calenders or roll mills for example.

Application of pressure and heat achieves a compression to $\leq 20\%$, preferably $\leq 15\%$, and especially $\leq 10\%$ of the original thickness.

25 As used herein, SAP refers to superabsorbent polymer. Superabsorbent polymer is capable of absorbing at least 10 times its own weight in 0.9% NaCl solution in the CRC test. SAP is known from the prior art and in this invention is preferably based on polyacrylate. The SAP can be present in any desired form, for example in particulate form, as a fiber, film or foam, particulate being preferred. Various SAPs and their production are described for example in WO 01/56625 30 page 3 line 37 to line 16 on page 19.

Fibers in the context of this invention comprehends all fibers which have been combined with SAP in the prior art. Preferred fibers are those which are present as a nonwoven. Preferred 35 fibers are described for example in WO 01/56625 page 19 line 40 to line 27 on page 20.

Pressing refers to the application of force to the surface of the material. This can be accomplished by means of classic presses, calenders or other suitable means.

The present invention is the first to provide a material which expands not less than 5-fold in one dimension and by less than 20% in the other two dimensions on addition of water (distilled water) or aqueous fluids (0.9% NaCl solution).

- 5 The material exhibits a substantially one-dimensional swelling performance on application of water or aqueous fluids. The expansion in water is generally not more than 20%, preferably not more than 18%, 16%, 14% or 12%, particularly preferably not more than 10%, 8%, 7%, or 6%, especially not more than 5%, 4%, 3%, 2% or even 1% in the x- and y-axes, while the material swells in the z-axis to more than 5-fold, 6-fold or 7-fold, preferably more than 8-fold or 9-fold,
10 especially more than 10-fold, 11-fold, 12-fold, 13-fold, 14-fold or even 15-fold or more.

The material after the application of pressure and heat is dimensionally stable; that is, little if any expansion takes place in the direction of the dimension in which the material was compressed.

- 15 The increase in the thickness of the material according to the present invention 60 days after compression is preferably less than 100%, preferably less than 80%, more preferably less than 60% and especially less than 50% based on the thickness directly after compression.

- The material according to the present invention is preferably obtainable by pressure and heat
20 treatment of material obtainable by in situ polymerization (as defined above and known from the prior art) of the SAP.

- The ultrathin materials according to the present invention are suitably producible using the webs recited in WO 01/56625. The SAP is preferably polymerized in situ on this web, but it is also
25 possible for example for materials to be compressed according to the present invention where the SAP has been sprinkled in or adhered atop the web.

- It is possible to add additives to the starting material before compression. This can be effected for example by spraying with the additive or an additive solution, by drenching and also by
30 sprinkling solid additives into the starting material or adhering solid additives atop the starting material. Additives refers for example to flavor and aroma chemicals, biocides and other odor-inhibiting materials, other active components, fertilizers and nutrients, dyes, surfactants, salts, polymers, softeners and others.

- 35 It is further possible to produce laminates (multilayer materials) according to the present invention. Possibilities include not only sandwich structures, where top and bottom layers of the same material enclose a second layer different than the top and bottom layers, but also structures having two or more (partly or partially) different layers.

For example, the starting material can be converted together with cotton webs, polyester webs, cotton-polyester blend webs, paper, paperboard into laminates by one-minute compression at 150°C and 80 bar, the starting material being simultaneously compressed and effective in
5 holding the components together.

By compression the present invention provides a material having a density of not less than 0.5 g/ccm, preferably not less than 0.6 g/ccm, more preferably not less than 0.7 g/ccm, even more preferably 0.8 g/ccm, yet more preferably not less than 0.9 g/ccm and especially up to not
10 less than 1 g/ccm or more. The maximum density is generally not more than 1.2 g/ccm. Pre-compression density is typically about 0.05 g/ccm. Since the material is flexible even after compression, the compressed material occupies a significantly smaller volume than the starting product.

15 Density refers to the weight of the material per unit volume, volume referring to the expanse of the material (length*width*thickness).

The ratio of teabag to retention in 0.9% NaCl solution is typically greater than 1.7 preferably greater than 1.9, more preferably greater than 2, especially greater than 2.2 (for granular SAP
20 the value is on the order 1.2 to 1.5). This high value indicates that the compressed material is capable of taking up larger amounts of water than granular SAP. This could be because the higher water uptake is due to the pores in the unfolding web structure (sponge effect). Following the rapid uptake of water, the water can subsequently be taken up by the SAP (water storage). The compressed material according to the present invention accordingly has a comparable
25 performance profile to fluff plus SAP.

The material according to the present invention has a retention in 0.9% NaCl solution of preferably more than 3 g/ccm, more preferably more than 5 g/ccm and especially more than
30 6.5 g/ccm or even 7 g/ccm.

Retention (even in the unit g/g) is higher for the pressed material than for the uncompressed, starting material. Compressing consequently leads to a material having better properties than are possessed by the starting material. The higher the temperature at which the compressing is carried out, the higher the retention value.

35 When the compressed material is placed in an atmosphere containing a high water vapor concentration, the material softens.

The material according to the present invention preferably has an FSEV after 60 seconds which is at least double that of the uncompressed material.

5 The material according to the present invention also preferably has an FSEV after 2 minutes which is at least 60% higher than that of the uncompressed material.

The material according to the present invention preferably has an EVUL after 60 seconds which is at least double that of the uncompressed material.

10 The material according to the present invention also preferably has an EVUL after 2 minutes which is at least 60% higher than that of the uncompressed material.

The AAP (0.7 psi) value of the material according to the present invention in 0.9% NaCl solution is preferably greater than 5 g/ccm or greater than 6.5 g/ccm, preferably greater than 9 g/ccm,
15 more preferably greater than 10 g/ccm, even more preferably greater than 11 g/ccm, especially greater than 12 g/ccm or even greater than 13 g/ccm.

The materials according to the present invention are also useful as a (multilayer) material to absorb water vapor. This property is reversible and survives a plurality of absorption-drying
20 cycles.

These materials are very useful inter alia in the following applications to absorb water or aqueous fluid, especially body fluid: hygiene articles (for example as absorbent core, storage and/or acquisition layer in infant diapers or adult incontinence articles, sanitary napkins, etc), bed
25 underlays, surgical drapes, wound contact materials, compresses, underlays for animal toilets, doormats, mats for absorbing snow, room climate improvement, climate improvement in seating and lying furniture, shoe soles and inserts, interlinings, garments, tablecloths, serviettes, cleaning cloths, seals or base material for seals, for example in the building sector, inner and outer pipe seals, cable sheaths, insulating and sealing material in the building industry, roof
30 membranes (water and vapor barriers), sealing membranes for landfills, flood control (building protection, tank systems), waterways, tunnel constructions, roadbuilding, pipe liner for drilling, siccatives for transportation and storage (cereals, for example), agricultural films, including erosion control, underlays for plants, encasement of root balls, mats for germinating seed material, mats for decorative plants (windowboxes etc), sealing material in the soil against rising
35 water and percolating water, decontamination of soils (removal of heavy metal salts for example), garbage bags, packaging, absorbent mats for transportation of moist and water-releasing goods, laminates, filters, fire protection.

The compressed material naturally has a smaller surface area than the starting material and hence a lower water take up rate. The absorption rate can be increased by increasing the surface area for example. Examples of suitable measures are roughening the surface or compressing in the presence of structure-conferring, surface-enlarging elements.

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Test results

The base material used was Luquafleece IS from BASF Aktiengesellschaft. It is typically possible to use nonwovens having a basis weight from 20 to 2000 g/m². The examples which follow utilize a PET nonwoven having a basis weight of 100 g/m² (Sawafill 8135 from Sandler). Luquafleece IS can be produced similarly to Example 9 of WO 01/56625 by loading the abovementioned nonwoven with 200 g/m² of SAP (100 g/m² each on both sides). It is also possible to use other loads, which are generally between 50 g/m² and 1000 g/m². The loads can be applied from one side or from both sides. Preference is given to loads between 100 g/m² and 300 g/m².

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A material was compressed under the conditions described in WO 01 / 56625 and tested for comparison. This material is referred to as "comparative material".

20 Samples produced at 5, 10, 80 and 160 bar and 50, 100, 150 and 200°C were each measured in 0.9% NaCl solution and also distilled water. The test results permit the following statements to be made:

Test methods whereby the materials according to the present invention differ most distinctly from Luquafleece and the compressed material produced according to WO 01 / 56625 are CRC and also test methods involving the unit "grams of absorbed fluid/ccm".

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Retention or CRC was measured as described in WO 01/56625 page 30 lines 40 ff.

30 AUL or AAP were measured as described in WO 01/56625 page 30 lines 16 ff. Teabag was determined in the same way as retention but without centrifugation.

Typical values for the material according to the present invention are reported hereinbelow, the parenthetical values being those of the uncompressed starting material and of the material compressed as per WO 01/56625 at lower temperatures.

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Retention (in 0.9% NaCl): 6 – 8.2 g/g (5.8 g/g & 5.9 g/g)

Retention (in 0.9% NaCl): 1500 - 2200 g/m² (1495 g/m² & 1346 g/m²)

- Retention (in 0.9% NaCl): 4 – 7.5 g/ccm (0.3 g/ccm & 0.4 g/ccm)
 Teabag (in 0.9% NaCl): 10 - 20 g/ccm (0.8 g/ccm & 1.3 g/ccm)
 Density: 0.5 – 1.2 g/ccm (0.05 g/ccm & 0.07 g/ccm)
 Expansion factor (in 0.9% NaCl): 10 – 21 (1.1 & 1.7)
- 5 AAP (in 0.9% NaCl; 0.7psi): 10.5 – 12.5 g/g (13 g/g & 12.3 g/g)
 AAP (in 0.9% NaCl; 0.7psi): 2500 - 3600 g/m² (3300 g/m² & 2968 g/m²)
 AAP (in 0.9% NaCl; 0.7psi): 6 – 14 g/ccm (0.6 g/g & 1.0 g/ccm)
- Retention (in distilled water): 13.5 – 19 g/g (13.3 g/g & 12.9 g/g)
- 10 Retention (in distilled water): 4000 - 5400 g/m² (3455 g/m² & 3106 g/m²)
 Retention (in distilled water): 10 – 16.5 g/ccm (0.7 g/ccm & 0.9 g/ccm)
 Teabag (in distilled water): 15 - 33 g/ccm (1.3 g/ccm & 3.0 g/ccm)
 Expansion factor (in distilled water): 15 – 33 (1.5 & 2.2)
 AAP (in distilled water): 18 – 22.5 g/g (18.9 g/g & 19.5 g/g)
- 15 AAP (in distilled water; 0.7psi): 5000 - 6200 g/m² (4750 g/m² & 4735 g/m²)
 AAP (in distilled water; 0.7psi): 10 – 22 g/ccm (0.9 g / g & 1.4 g/ccm)

- The nonwoven without SAP exhibits fundamentally different properties than Luquafleece IS. There are even bigger differences between compressed nonwoven and compressed
- 20 Luquafleece IS. The pure nonwoven has a high teabag value of 37 g/g in NaCl owing to the sponge effect and since the salt content is immaterial to this effect the same value in distilled water at 38 g/g. For the same reason the CRC value is extremely low in both the media mentioned, at 0.4 g/g; this is because the centrifuging removes substantially all of the liquid from the web.
- 25
- When the nonwoven is compressed by the application of pressure and heat, it does not swell in the presence of water. The teabag and CRC values are correspondingly low. A nonwoven which had been pressed at 200°C and 80 bar for 1 minute exhibited a teabag value of 1.9 g/g and a CRC value of 0.3 g/g in 0.9% NaCl. The thickness of the pressed material remained unchanged
- 30 at 0.1 mm. These results show that swelling in the z-axis is only possible in the presence of SAP.

- Ultrathin materials according to the present invention can in principle also be produced by sprinkling SAP granules or powder into a nonwoven, optionally fixing the SAP to the nonwoven
- 35 using an adhesive or anchoring it to the web by way of other techniques. Although the physical properties are similar to those of the pressed in situ material, products which have been reversibly fixed on the nonwoven have the disadvantage that the SAP will become detached again from the nonwoven by the swelling in the presence of water. In the case of compressed

materials which have been polymerized in situ onto fibers, by contrast, the SAP is firmly attached to the fiber and does not become detached again even in the presence of water.

Dimensional stability

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The compressed material is dimensionally stable; that is, the material expands insignificantly, if at all, even in the course of prolonged storage at room temperature and relative humidities of preferably less than 60%. This dimensional stability was found with all samples which were compressed at a temperature of more than 60°C and a pressure of more than 5 bar. In the case

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of the comparative material produced according to WO 01 / 56625, in contrast, an expansion of the material took place under the abovementioned conditions:

| | Sample [mm] | Thickness directly after compression [mm] | Thickness after 60 days |
|----|-------------|---|-------------------------|
| | 1 | 0.8 | 2.4 |
| 15 | 2 | 0.7 | 1.8 |
| | 3 | 0.7 | 1.9 |
| | 4 | 0.8 | 2.3 |

Free-swell expansion volume as a function of time

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This parameter is described in WO 01 / 56625 at page 31 lines 33 ff – FSEV. An SAP loading of 200 g/m² produced the following values in 0.9% NaCl:

| | | |
|----|-------------|--------|
| | 5 seconds | 1.4 ml |
| | 10 seconds | 2.2 ml |
| 25 | 30 seconds | 4.3 ml |
| | 60 seconds | 5.9 ml |
| | 120 seconds | 7.3 ml |
| | 300 seconds | 8.5 ml |
| | 600 seconds | 9.0 ml |

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Compressed materials which were compressed at various pressures and temperatures (duration: 1 minute) gave the following measured values. "Comparison" refers to the sample which was produced according to WO 01 / 56625 (5 bar, 48 s, 50°C).

35 Determination of free-swell expansion volume (FSEV)

| Time | 5bar 100°C | 10bar 100°C | 80bar 100°C | 5bar 150°C | 10bar 150°C | 80bar 150°C | Comparison |
|---------|---------------|----------------|----------------|---------------|----------------|----------------|------------|
| 10 sec | 2.4 ml | 2.4 ml | 2.2 ml | 1.3 ml | 2.2 ml | 0.1 ml | 2.4 ml |
| 30 sec | 6.8 ml | 7.1 ml | 7.5 ml | 5.7 ml | 6.4 ml | 0.7 ml | 5.8 ml |
| 60 sec | 8.7 ml | 9.3 ml | 10.1 ml | 9.0 ml | 9.3 ml | 4.2 ml | 7.5 ml |
| 120 sec | 9.8 ml | 10.5 ml | 11.1 ml | 10.7 ml | 10.8 ml | 10.1 ml | 8.7 ml |
| 300 sec | 10.4 ml | 11.3 ml | 11.8 ml | 11.5 ml | 11.6 ml | 11.5 ml | 9.5 ml |
| 600 sec | 10.6 ml | 11.5 ml | 12.0 ml | 11.7 ml | 11.8 ml | 11.8 ml | 9.9 ml |

- The data show that the FSEV values of the material according to the present invention (with the exception of 80 bar/150°C) are distinctly higher than those of the compressed material described in WO 01 / 56625 after just 30-60 seconds. The data also show that the final value is almost
- 5 reached after about 300 seconds.

When the measurement is performed by determining the expansion volume under a load of 0.5 psi (EVUL as per WO 01/56625 page 32 lines 6 ff), a similar picture is obtained:

- 10 Determination of expansion volume under load (EVUL) at 0.5 psi

| Time | 5bar 100°C | 10bar 100°C | 80bar 100°C | 5bar 150°C | 10bar 150°C | 80bar 150°C | Comparison |
|---------|---------------|----------------|----------------|---------------|----------------|----------------|------------|
| 10 sec | 0.9 ml | 0.4 ml | 1.2 ml | 0.6 ml | 0.8 ml | 0.1 ml | 0.1 ml |
| 30 sec | 3.0 ml | 2.2 ml | 3.7 ml | 3.0 ml | 3.1 ml | 1.4 ml | 1.0 ml |
| 60 sec | 4.4 ml | 3.6 ml | 5.1 ml | 4.7 ml | 4.7 ml | 4.2 ml | 2.1 ml |
| 120 sec | 5.0 ml | 4.6 ml | 5.8 ml | 6.0 ml | 5.8 ml | 5.8 ml | 3.1 ml |
| 300 sec | 5.6 ml | 5.3 ml | 6.1 ml | 6.6 ml | 6.4 ml | 6.4 ml | 3.8 ml |
| 600 sec | 5.6 ml | 5.7 ml | 6.3 ml | 6.9 ml | 6.6 ml | 6.4 ml | 3.8 ml |

- The samples produced according to the present invention are faster than the comparative sample in water takeup under pressure of 0.5 psi. Only the sample produced at 80°C/150 bar gives the same value after 10 seconds, but here too all the other measured results are better
- 15 than with the comparative sample.

Absorption of water vapor

- 20 The compressed material is capable of absorbing significant amounts of water vapor and rereleasing them at low relative humidities (comparable properties to Luquafl fleece IS). Various specimens (each 100 x 100 mm) which were compressed for one minute at 160 bar and

- different temperatures were stored in a desiccator at room temperature and 95% relative humidity for 24 h. The specimens were then dried in air at 23°C and 45% relative humidity. The results are discernible from Table A. Subsequently, the specimens were again stored in a desiccator at 95% relative humidity for 24 h and thereafter again dried at room temperature and 45% relative humidity. This cycle was carried out a third time. The results after the third cycle are reported in Table B.

Table A

| Sample | Moisture absorption Weight after 24 h (in % of starting weight) | Moisture absorption Weight after 24 h (in % of starting weight) |
|--------------------|---|---|
| Luquafleece IS | 143 | 107 |
| P1 (50°C/160bar) | 142 | 107 |
| P2 (100°C/160bar) | 144 | 107 |
| P3 (150°C/160bar) | 149 | 112 |
| P4 (200°C/160 bar) | 149 | 114 |

10 Table B

| Sample | Moisture absorption Weight after 3 cycles (in % of starting weight) | Moisture absorption Weight after 3 cycles (in % of starting weight) |
|-------------------|---|---|
| Luquafleece IS | 149 | 107 |
| P1 (50°C/160bar) | 152 | 107 |
| P2 (100°C/160bar) | 152 | 109 |
| P3 (150°C/160bar) | 157 | 113 |
| P4 (200°C/160bar) | 155 | 114 |

Examples

The tests were carried out on samples measuring 30 x 50 mm in area. The AAP tests were carried out using a circular piece 6 cm in diameter (area 28.3 cm²). The tests were carried out at 0.7 psi.

The 30 x 50 mm samples were observed to swell essentially in the z-direction. A swelling of about 10% was measured in the x- or y-direction, the third dimension remained unchanged (the web is more easily extendable in one axis than in the other dimension).

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All tests were carried out according to the commonly used standard methods for SAP.

Inventive example 1

Luquafleece IS was compressed at 100°C and 160 bar for one minute. 30 x 50 mm samples were measured. The following data were obtained in distilled water:

Thickness of compressed material: 0.3 mm

CRC: 15.6 g / g

CRC: 4905 g / m²

20 Teabag value: 30.6 g / ccm*)

Retention: 16.4 g / ccm*)

Density of material used: 0.984 g / ccm

Expansion factor in z-axis: 31.0

AAP (0.7 psi): 19.1 g / g

25 AAP (0.7 psi): 5689 g / m²

AAP (0.7 psi): 19.0 g / ccm

*) the reported value refers to the material used

30 Inventive example 2

Luquafleece IS was compressed at 100°C and 80 bar for one minute. 30 x 50 mm samples were measured. The following data were obtained in distilled water:

Thickness of compressed material: 0.3 mm

35 CRC: 15.7 g / g

CRC: 4592 g / m²

Teabag value: 31.7 g / ccm*)

Retention: 15.3 g / ccm*)

Density of material used: 1.042 g / ccm

Expansion factor in z-axis: 28.7

AAP (0.7 psi): 19.8 g / g

AAP (0.7 psi): 5477 g / m²

5 AAP (0.7 psi): 18.2 g / ccm

*) the reported value refers to the material used

Inventive example 3

10 Luquafleece IS was compressed at 150°C and 80 bar for one minute. 30 x 50 mm samples were measured. The following data were obtained in distilled water:

Thickness of compressed material: 0.25 mm

CRC: 14.6 g / g

15 CRC: 3326 g / m²

Teabag value: 32.9 g / ccm*)

Retention: 16.6 g / ccm*)

Density of material used: 0.969 g / ccm

Expansion factor in z-axis: 32.5

20 AAP (0.7 psi): 19.2 g / g

AAP (0.7 psi): 4382 g / m²

AAP (0.7 psi): 21.9 g / ccm

*) the reported value refers to the material used

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Inventive example 4

Luquafleece IS was compressed at 200°C and 80 bar for one minute. 30 x 50 mm samples were measured. The following data were obtained in distilled water:

30 Thickness of compressed material: 0.25 mm

CRC: 18.4 g / g

CRC: 4577 g / m²

Teabag value: 30.0 g / ccm*)

Retention: 15.3 g / ccm*)

35 Density of material used: 0.959 g / ccm

Expansion factor in z-axis: 28.0

AAP (0.7 psi): 22.4 g / g

AAP (0.7 psi): 6184 g / m²

AAP (0.7 psi): 20,6 g / ccm

*) the reported value refers to the material used

5 Inventive example 5

Luquafleece IS was compressed at 150°C and 10 bar for one minute. 30 x 50 mm samples were measured. The following data were obtained in distilled water:

Thickness of compressed material: 0.4 mm

10 CRC: 16.1 g / g

CRC: 4830 g / m²

Teabag value: 24.5 g / ccm*)

Retention: 12.1 g / ccm*)

Density of material used: 0.697 g / ccm

15 Expansion factor in z-axis: 22.3

AAP (0.7 psi): 20.9 g / g

AAP (0.7 psi): 6042 g / m²

AAP (0.7 psi): 15.1 g / ccm

20 *) the reported value refers to the material used

Comparative example 1 (Luquafleece IS)

Thickness of Luquafleece IS: 5.3 mm

CRC: 13.3 g / g

25 CRC: 3455 g / m²

Teabag value: 1.3 g / ccm*)

Retention: 0.73 g / ccm*)

Density of material used: 0.051 g / ccm

Expansion factor in z-axis: 1.5

30 AAP (0.7 psi): 18.9 g / g

AAP (0.7 psi): 4750 g / m²

AAP (0.7 psi): 0.9 g / ccm

*) the reported value refers to the material used

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Comparative example 2 (Luquafleece IS compressed at 6 bar and 50°C for 48 s)

Thickness of compressed material: 3.1 mm

CRC: 12.9 g / g

CRC: 3106 g / m²

Teabag value: 2.0 g / ccm*)

Retention: 0.9 g / ccm*)

Density of material used: 0.074 g / ccm

5 Expansion factor in z-axis: 2.2

AAP (0.7 psi): 19.5 g / g

AAP (0.7 psi): 4735 g / m²

AAP (0.7 psi): 1.4 g / ccm

10 *) the reported value refers to the material used

Increasing the pressure at low temperatures does not result in significant improvement. This is to be documented by means of the following example:

15 Comparative example 3 (Luquafl fleece IS 60 compressed at 80 bar and 50°C for 60 s)

Thickness of compressed material: 0.8 mm

Thickness of compressed material after 4 weeks: 1.2 mm

CRC: 12.5 g / g

CRC: 2730 g / m²

20 Teabag value: 3.0 g / ccm*)

Retention: 1.9 g / ccm*)

Density of material used: 0.159 g / ccm

Expansion factor in z-axis: 4.6

AAP (0.7 psi): 18.4 g / g

25 AAP (0.7 psi): 4276 g / m²

AAP (0.7 psi): 3.1 g / ccm

*) the reported value refers to the material used

30 Inventive example 6

Luquafl fleece IS was compressed at 100°C and 160 bar for one minute. 30 x 50 mm samples were measured. The following data were obtained in 0.9% NaCl:

Thickness of compressed material: 0.3 mm

35 CRC: 6.5 g / g

CRC: 2069 g / m²

Teabag value: 19.4 g / ccm*)

Retention: 6.9 g / ccm*)

Density of material used: 0.977 g / ccm

Expansion factor in z-axis: 19.0

AAP (0.7 psi): 10.6 g / g

AAP (0.7 psi): 3357 g / m²

5 AAP (0.7 psi): 11.2 g / ccm

*) the reported value refers to the material used

Inventive example 7

10 Luquafleece IS was compressed at 100°C and 80 bar for one minute. 30 x 50 mm samples were measured. The following data were obtained in 0.9% NaCl:

Thickness of compressed material: 0.3 mm

CRC: 7.3 g / g

15 CRC: 2151 g / m²

Teabag value: 19.5 g / ccm*)

Retention: 7.2 g / ccm*)

Density of material used: 1.073 g / ccm

Expansion factor in z-axis: 19.3

20 AAP (0.7 psi): 11.3 g / g

AAP (0.7 psi): 3039 g / m²

AAP (0.7 psi): 10.1 g / ccm

*) the reported value refers to the material used

25

Inventive example 8

Luquafleece IS was compressed at 150°C and 160 bar for one minute. 30 x 50 mm samples were measured. The following data were obtained in 0.9% NaCl:

30 Thickness of compressed material: 0.25 mm

CRC: 6.4 g / g

CRC: 1489 g / m²

Teabag value: 16.5 g / ccm*)

Retention: 7.4 g / ccm*)

35 Density of material used: 1.113 g / ccm

Expansion factor in z-axis: 20.0

AAP (0.7 psi): 11.2 g / g

AAP (0.7 psi): 2650 g / m²

AAP (0.7 psi): 13.3 g / ccm

*) the reported value refers to the material used

5 Inventive example 9

Luquaflleece IS was compressed at 150°C and 80 bar for one minute. 30 x 50 mm samples were measured. The following data were obtained in 0.9% NaCl:

Thickness of compressed material: 0.25 mm

10 CRC: 6.5 g / g

CRC: 1490 g / m²

Teabag value: 20.1 g / ccm*)

Retention: 7.5 g / ccm*)

Density of material used: 1.006 g / ccm

15 Expansion factor in z-axis: 20.5

AAP (0.7 psi): 11.7 g / g

AAP (0.7 psi): 2792 g / m²

AAP (0.7 psi): 14.0 g / ccm

20 *) the reported value refers to the material used

Inventive example 10

Luquaflleece IS was compressed at 150°C and 10 bar for one minute. 30 x 50 mm samples were measured. The following data were obtained in 0.9% NaCl:

25

Thickness of compressed material: 0.4 mm

CRC: 6.9 g / g

CRC: 2128 g / m²

Teabag value: 10.5 g / ccm*)

30 Retention: 4.3 g / ccm*)

Density of material used: 0.590 g / ccm

Expansion factor in z-axis: 11.2

AAP (0.7 psi): 12.3 g / g

AAP (0.7 psi): 3251 g / m²

35 AAP (0.7 psi): 6.5 g / ccm

*) the reported value refers to the material used

Inventive example 11

Luquafleece IS was compressed at 200°C and 160 bar for one minute. 30 x 50 mm samples were measured. The following data were obtained in 0.9% NaCl:

- 5 Thickness of compressed material: 0.22 mm
CRC: 8.2 g / g
CRC: 2230 g / m²
Teabag value: 15.5 g / ccm*)
Retention: 7.4 g / ccm*)

- 10 Density of material used: 1.043 g / ccm
Expansion factor in z-axis: 18.9
AAP (0.7 psi): 11.8 g / g
AAP (0.7 psi): 3251 g / m²
AAP (0.7 psi): 10.9 g / ccm

- 15 *) the reported value refers to the material used

Comparative example 4 (Luquafleece IS)

- Thickness of Luquafleece IS: 5.2 mm
- 20 CRC: 5.8 g / g
CRC: 1495 g / m²
Teabag value: 0.8 g / ccm*)
Retention: 0.3 g / ccm*)
Density of material used: 0.047 g / ccm
- 25 Expansion factor in z-axis: 1.1
AAP (0.7 psi): 13.0 g / g
AAP (0.7 psi): 3300 g / m²
AAP (0.7 psi): 0.6 g / ccm

- 30 *) the reported value refers to the material used

Comparative example 5 (Luquafleece IS compressed at 6 bar and 50°C for 48 s)

- Thickness of compressed material: 3.1 mm
- 35 CRC: 5.9 g / g
CRC: 1346 g / m²
Teabag value: 1.3 g / ccm*)
Retention: 0.4 g / ccm*)
Density of material used: 0.072 g / ccm

Expansion factor in z-axis: 1.7

AAP (0.7 psi): 12.3 g / g

AAP (0.7 psi): 2968 g / m²

AAP (0.7 psi): 1.0 g / ccm

5

*) the reported value refers to the material used

Increasing the pressure at low temperatures does not result in significant improvement. This is to be documented by means of the following example:

10

Comparative example 6 (Luquafl fleece IS is compressed at 80 bar and 50°C for 60 s)

Thickness of compressed material: 0.8 mm

Thickness of compressed material after 4 weeks: 1.2 mm

CRC: 5.8 g / g

15

CRC: 1330 g / m²

Teabag value: 1.0 g / ccm*)

Retention: 0.3 g / ccm*)

Density of material used: 0.184 g / ccm

Expansion factor in z-axis: 2.8

20

AAP (0.7 psi): 11.7 g / g

AAP (0.7 psi): 2686 g / m²

AAP (0.7 psi): 3.0 g / ccm

*) the reported value refers to the material used

25

Influencing the hardness of the ultrathin material

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Hardness and moisture absorption rate can be influenced for example by adding softening chemicals or by structuring (enlarging) the surface area of the ultrathin material. Examples of useful softening chemicals are tertiary alkanolamines. The free acid groups of the SAP are preferably at least 20 mol% neutralized. Preferred alkanolamines are selected from the group consisting of triethanolamine, methyldiethanolamine, dimethylaminodiglycol, dimethylethanolamine, N,N,N',N'-tetra(hydroxyethyl)ethylenediamine. These possibilities are not new per se, but are new in connection with influencing the water absorption capacity of superabsorbent sheetlike structures. In addition, the ultrathin material can be made softer by a specifically targeted addition of small amounts of water. The softening possibilities mentioned can also be combined. To achieve a very homogeneous increase in moisture content, the

35

material is preferably treated with water vapor or water mist. To preserve the softness, however, it is subsequently necessary to pack the material airtight.

5 The change in hardness can be measured for example using an apparatus with which a sphere is pressed into the material for a defined distance while the force required for this distance is measured. The ultrathin material was placed in a saturated water vapor atmosphere at room temperature for 24 h. In the course of this period, the material absorbed 70% of its own weight in moisture. After 24 h, the sample was taken out of this atmosphere and the force needed to press the sphere 10 mm deep into the material was measured. The measurement was repeated
10 in an hourly rhythm. The material was situated for this period in an environment of 50% relative humidity at 24°C; that is, the moisture content decreased steadily in this period and had almost returned back to the starting value after 7 h.

The following moisture contents (based on the material used prior to moist storage) were
15 measured:

70% after 24 h in saturated water vapor atmosphere

40% after 1 h at 24°C and 50% relative humidity

11% after 3 h at 24°C and 50% relative humidity

1% after 7 h at 24°C and 50% relative humidity

20

The materials used for the hardness measurement had been compressed at 160 bar and different temperatures for 1 minute. The following measurements (10 mm path length) were obtained.

25 Sample 1 (200°C / 160 bar):

Force: 8 N measurement instantly, 10 N after 1 h, 21 N after 3 h, 33 N after 7 h

Sample 2 (150°C / 160 bar):

Force: 7 N measurement instantly, 9 N after 1 h, 18 N after 3 h, 24 N after 7 h

Sample 3 (50°C / 160 bar):

30 Force: 8 N measurement instantly, 11 N after 1 h, 16 N after 3 h, 19 N after 7 h

Comparison: Luquafleece IS

Force: 4 N measurement instantly, 4.5 N after 1 h, 6 N after 3 h, 10 N after 7 h

35 It is clearly visible that the materials harden as the moisture content decreases. The measured values further reveal that approximately the same force has to be applied to press the sphere 10 mm into a dry Luquafleece as into an ultrathin material having a moisture content of 70%.